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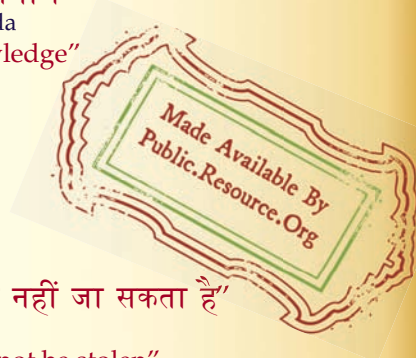
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“Knowledge is such a treasure which cannot be stolen”

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IS : 1560 (Part I) - 1974

(Reaffirmed 2004)

Indian Standard
METHOD FOR
ESTIMATION OF CARBOXYLIC ACID GROUPS
IN CELLULOSIC TEXTILE MATERIALS

PART I IODOMETRIC METHOD

(First Revision)

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METHOD FOR ESTIMATION OF CARBOXYLIC ACID GROUPS IN CELLULOSIC TEXTILE MATERIALS

PART I IODOMETRIC METHOD*(First Revision)*

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Indian Standard

METHOD FOR ESTIMATION OF CARBOXYLIC ACID GROUPS IN CELLULOSIC TEXTILE MATERIALS

PART I IODOMETRIC METHOD

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part I) (First Revision) was adopted by the Indian Standards Institution on 4 October 1974, after the draft finalized by the Chemical Methods of Test Sectional Committee had been approved by the Textile Division Council.

0.2 In the cellulosic textile industry, cellulose in the form of fibres, yarn and fabric comes in contact with different oxidizing agents during the various chemical processing treatments. The action of these oxidizing agents on cellulose may result in the formation of oxycelluloses of acidic character attributable to the introduction of carboxyl groups into the cellulose chain molecule. Purified cotton cellulose, not subjected to any treatment with oxidizing agents, also behaves as though it possesses a very small content of carboxylic acid groups. The absorption of metallic ions from aqueous solutions of their salts is an outstanding property of oxycelluloses. This property in celluloses and oxycelluloses is due to the presence of carboxylic acid groups in them. The estimation of carboxylic acid groups present in cellulosic textile materials is a method of determining the extent of this type of oxidation of cellulose. This is a useful supplementary test to other tests such as copper number and fluidity tests.

0.3 This standard, first published in 1962, is now being issued in its revised version as Part I. The second part of the standard is being issued to cover the sodium chloride — sodium bicarbonate method as an alternative one for the estimation of carboxyl acid group content of cellulosic textile materials.

0.3.1 In this revision, besides other modifications, the reaction time has been reduced from 24 hours at room temperature to 1 hour at 60°C (see 7.2).

0.4 Considerable assistance has been derived from the following publications :

Achwal (W B), Nabar (G M) and Padmanabhan (C V). Estimation of carboxylic acid groups in cellulosic materials. *J Sci Industr Res.* 17BC; 1958; 497.

Nabar (G M) and Padmanabhan (C V). Estimation of carboxyl groups in cellulosic materials. *Pr Ind Acad Sci.* 31A; 1950; 371.

0.5 In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part I) prescribes the iodometric method for estimating carboxylic acid groups in cellulosic textile materials.

2. PRINCIPLE

2.1 Cation-free cellulosic material is suspended in potassium iodide-potassium iodate-sodium chloride solution to which sodium thiosulphate is added to prevent the loss of iodine due to side reactions and vaporization as also to facilitate the completion of the reaction by removal of the iodine liberated from the sphere of reaction. At the end of the requisite period, the excess of thiosulphate is titrated back with standard iodine solution, and the amount of thiosulphate consumed indicates the extent of carboxyl content.

3. SAMPLING

3.1 Sample shall be so selected as to be representative of the lot.

3.1.1 Sample drawn in accordance with the procedure laid down in the relevant material specification or in compliance with an agreement between the buyer and the seller shall be held to be representative of the lot.

4. PREPARATION OF TEST SPECIMENS

4.1 Cut the sample under test into small pieces. Mix all the pieces thoroughly and draw at least two specimens, each weighing about 2 g.

5. APPARATUS

5.1 Automatic Burette — of 50 ml capacity.

5.2 Erlenmeyer Flask — of Pyrex glass (or similar heat-resistant glass), fitted with a glass stopper and of 250 ml capacity.

5.3 Small Compressor, Vacuum Pump or Water Jet Pump — for blowing air; capable of producing a pressure sufficient to stir the liquid in the flask during titration.

*Rules for rounding off numerical values (*revised*).

6. REAGENTS

6.1 Quality of Reagents

6.1.1 Unless specified otherwise, pure chemicals shall be employed in the test and distilled water (*see* IS : 1070-1960*) shall be used where the use of water as reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

6.1.2 All reagents shall be prepared using carbon dioxide-free distilled water which may be obtained by the method prescribed in Appendix A. All reagents shall be stored in containers with soda-lime traps in order to protect the reagents from coming into contact with atmospheric carbon dioxide.

6.2 The reagents required for the test shall be as given below.

6.2.1 Potassium Iodide — Potassium Iodate — Sodium Chloride Solution — Prepare the solution by dissolving the following reagents in the quantities indicated against each in carbon dioxide-free distilled water to make 2 litres :

Potassium iodide (analytical grade)	83.0
Potassium iodate (analytical grade)	21.4
Sodium chloride (analytical grade)	50.0
Sodium thiosulphate (analytical grade)	5.0

6.2.2 Standard Sodium Thiosulphate Solution — 0.02 N.

6.2.3 Iodine Solution — 0.02 N. Dissolve 2.6 g of iodine and 4 g of potassium iodide in about 25 ml of water by warming gently. Dilute the solution to 1 litre. Standardize against standard thiosulphate solution.

6.2.4 Starch Solution — 0.2 percent (m/v).

6.2.5 Hydrochloric Acid Solution — approximately 0.5 N.

7. PROCEDURE

7.1 Take one test specimen and steep it for 2 hours in 0.5 N hydrochloric acid at room temperature, keeping the material to liquor ratio as 1 : 50. Wash the specimen on a suction filter thoroughly with distilled water till the washings are neutral (as indicated by bromocresol purple). Dry the specimen in air.

NOTE — The above treatment renders the specimen cation-free.

7.2 Transfer from the automatic burette 50 ml of the solution prepared as in 6.2.1 to the Erlenmeyer flask. Weigh accurately 0.5 to 1.0 g of air-dry cation-free specimen (*see* 7.1) and transfer it to the flask. Stopper the flask,

*Specification for water, distilled quality (*revised*). (Since revised).

shake it gently to mix the contents thoroughly and immerse it in a water-bath maintained at $60 \pm 1^\circ\text{C}$ for 1 hour. Cool the flask quickly and titrate the excess of thiosulphate solution against 0.02 N iodine solution using starch as indicator. Bubble by means of a compressor or a vacuum pump or a water jet pump carbon dioxide-free air through the mixture throughout the titration.

7.3 Carry out a blank titration following the same procedure (*see 7.2*) but without the test specimen.

7.4 Determine the moisture content of the cation-free material (*see 7.1*) separately as given in IS : 199-1973* and calculate the oven-dry mass of the cation-free specimen taken for the test (*see 7.2*).

7.5 Repeat the test with the remaining test specimen(s).

8. CALCULATION

8.1 Calculate the carboxylic acid group content of each specimen, in milli-equivalents of $-\text{COOH}$ per 100 g, of the sample, by the following formula :

$$\text{Carboxylic acid group content, expressed as milli-equivalents of } -\text{COOH per 100 g of sample} = \frac{(V_1 - V_2) \times N \times 100}{M}$$

where

V_1 = quantity in millilitres of iodine solution required for the blank (*see 7.3*),

V_2 = quantity in millilitres of iodine solution required for actual test (*see 7.2*),

N = normality of iodine solution, and

M = oven-dry mass in gram of the cation-free specimen taken for the test (*see 7.4*).

8.2 Calculate the average of the values obtained as in 8.1.

9. REPORT

9.1 The report shall include the following :

- a) Carboxylic acid group content, and
- b) Number of specimens tested.

*Methods for estimation of moisture, total size or finish, ash and fatty matter in grey and finished cotton textile materials (*second revision*).

APPENDIX A

(Clause 6.1.2)

METHOD FOR PREPARATION OF CARBON DIOXIDE FREE DISTILLED WATER

A-1. APPARATUS

A-1.1 Flask — of hard glass, fitted with a stopper and of required capacity.

A-1.2 Nessler Tube — of thin colourless glass, with flat bottom, about 25 mm in diameter and about 150 mm in length.

A-2. REAGENT

A-2.1 Saturated Calcium Hydroxide Solution

A-3. PROCEDURE

A-3.1 Boil the required amount of distilled water in the flask for about 5 minutes.

A-3.2 Transfer 25 ml of the boiled distilled water to the Nessler tube. Add to it 25 ml of saturated calcium hydroxide solution and mix well. After 5 minutes, observe whether there is any turbidity or precipitate.

A-3.3 Take the distilled water to be free from carbon dioxide if no turbidity or precipitate is observed (see A-3.4). Stopper the flask and cool the content.

A-3.4 If, however, turbidity or precipitate is observed, repeat as many times as may be necessary the procedure prescribed in A-3.1 and A-3.2.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	Unit	Symbol	Conversion
Force	newton	N	1 N = 0.101 972 kgf
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m ²
Frequency	hertz	Hz	1 Hz = 1 c/s (s ⁻¹)
Electric conductance	siemens	S	1 S = 1 A/V
Pressure, stress	pascal	Pa	1 Pa = 1 N/m ²

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